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The Performance of Perfluoropolyalkyethers Under Boundary Conditions

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Prepared for

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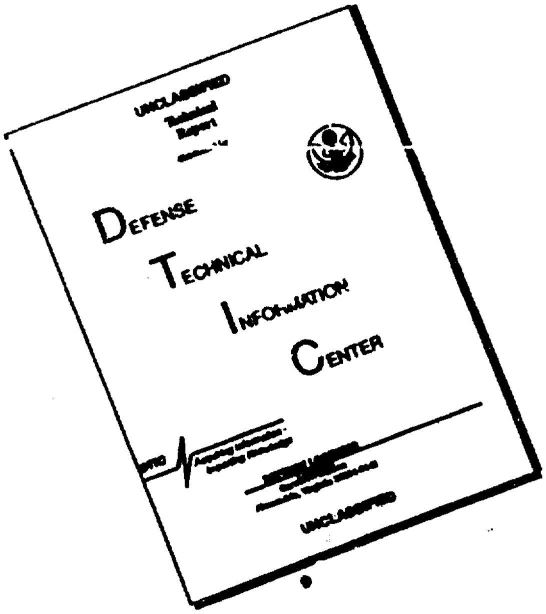

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Perfluoropolyalkylethers (PFPEs) are a relatively recent addition to the family of lubricants used in spacecraft applications. They have excellent properties but do not perform well under boundary lubrication conditions. This has been demonstrated in testing under continuous rolling and oscillatory motion. The PFPEs degrade under conditions in which reactive iron metal is exposed, leading to catalytic reactions. Improved performance can be achieved through the use of soluble antiwear additives, which are currently being developed. Improved performance can also be achieved through the prevention of metallic iron exposure by using ceramic hard-coated and solid ball-bearing components.

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ORBITAL ENVIRONMENT PERSPECTIVE

The current trend in spacecraft design is to attempt to significantly extend on-orbit lifetimes to 10 years or longer. This trend places severe requirements on the reliability of spacecraft systems, including moving mechanical assemblies. The shuttle orbiter can be employed for limited servicing of spacecraft in low-earth orbit, but there are no service stations at higher orbital altitudes. Thus, satellites at geosynchronous altitudes will be required to travel in excess of 200,000,000 miles without an oil change. Not only will the normal pathways associated with lubricant degradation need to be reduced or alleviated, but the effects of the orbital environment will have to be handled.

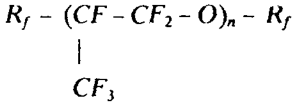
The orbital environment is not benign. Solar electromagnetic and particle radiation can result in bond breaking and degradation in organic molecules,[1,2] collisions with atomic oxygen in low-earth orbit can significantly alter lubricants,[3] the ambient temperature can be extreme, and low ambient pressures can result in loss of lubricant by evaporative transport. Lubricants that will meet these challenges must have special physical and chemical properties.

PERFLUOROPOLYALKYLETHER LUBRICANTS

A recent addition to the group of lubricants used for spacecraft applications is the family of synthetic perfluoropolyalkylether (PFPE) oils and oil-based greases. These oils and greases are being used in an increasing variety of applications. They have some outstanding physical and chemical properties, including a high degree of chemical stability, large viscosity index, good lubricity, and nonflammability.

The commercially available PFPE materials come in three different structural types:

$$R_f - (CF_2 - CF_2 - O)_x - (CF_2 - O)_y - R_f \qquad \text{(I)}$$



$$R_f - (\underset{\displaystyle CF_3}{\underset{|}{CF}} - CF_2 - O)_n - R_f \qquad \text{(II)}$$

$$R_f - (CF_2 - CF_2 - CF_2 - O)_n - R_f \qquad \text{(III)}$$

where R_f is a fluorinated end group of unspecified length. Structure (I) corresponds to the Fomblin Z fluid that is used in the Braycote line of PFPE lubes, (II) represents the Fomblin Y and Krytox fluids, and (III) is representative of the Demnum fluids.

EXPERIENCE UNDER BOUNDARY CONDITIONS

Despite the low chemical reactivity of the PFPE fluids, which is not unlike that of Teflon-coated cookware, these lubricants have exhibited failures very early in life under boundary lubrication conditions. The following examples illustrate these failures.

In a test to determine lift-off speed for a certain application, we observed what appeared to be friction polymer formation in the raceways when a PFPE grease was used. The polymeric, fluorocarbon coating was removed to reveal significant pitting of the raceway surface, apparently due to chemical "milling" of the surface metal. Figure 1 shows scanning electron micrographs of an unused raceway, of the area under the polymeric coating in the raceway, and of an area outside the contact zone. It is clear that chemical reaction had occurred and that the wear track area was covered by the polymeric material.[4]

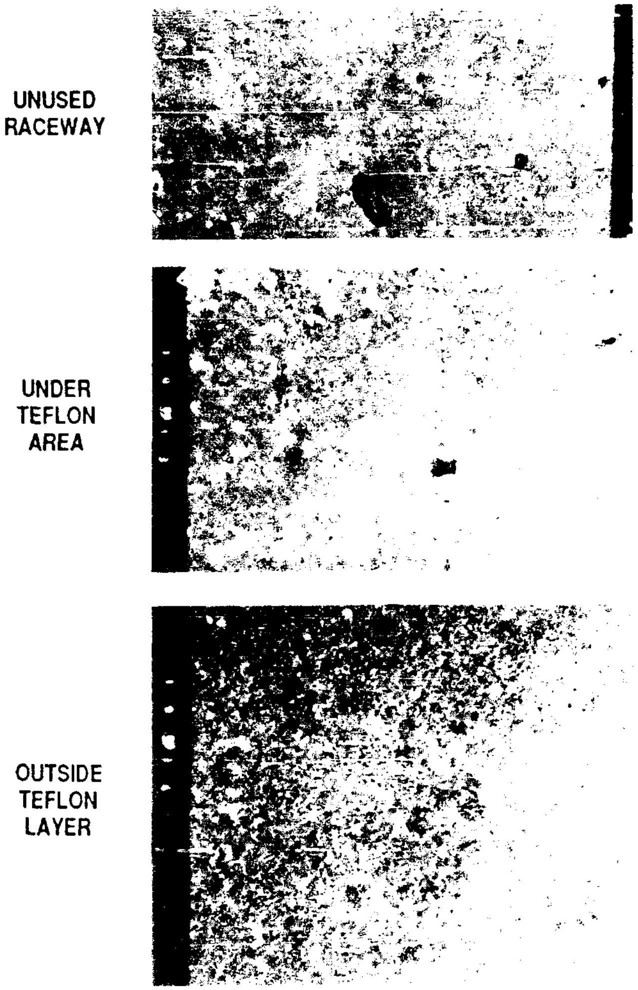


Figure 1. Scanning electron micrographs of bearing raceway surfaces.

In another test of a PFPE grease under boundary conditions, we discovered that ~2% of a formulated hydrocarbon oil present as a contaminant resulted in superior performance.[5] Figure 2 shows a plot of total revolutions to failure vs Hertzian stress in the contact. The contaminated grease clearly outperformed the uncontaminated grease. We believe that the antiwear additives in the hydrocarbon oil led to the superior performance of the grease. The same effect has been reported by other researchers.[6]

Another example of inadequate boundary performance is illustrated in Figure 3. In this figure, total revolutions to failure are plotted for a series of lubricants run under identical boundary lubrication conditions.[5] Samples 1 and 2 are formulated petroleum-based oils, samples 3–7 are formulated poly-α-olefin (PAO) synthetic hydrocarbon lubricants, and sample 8 is a branched PFPE with structure (II). The PFPE exhibits the poorest performance.

Poor performance was also observed in a laboratory test that simulated an oscillatory spacecraft mechanism. Small R2 ball bearings were lubricated with either a linear PFPE fluid, a chloroalkylsiloxane (CAS) oil, or a PAO oil.[7] The PFPE and CAS oils failed at ~2000 h. Figure 4 gives a plot of torque vs time for one pair of PFPE-lubricated bearings. (The "sinusoidal" variation in torque is due to the oscillatory motion in the test.) After 2350 h, the torque is essentially zero, indicating that the preload had been completely relieved through wear of the contacting members. Figure 5 shows the one PFPE-lubricated bearing pair after testing. It is clear that significant lubricant degradation has occurred. In contrast, Figure 6 shows the torque trace for one of the PAO-lubricated bearing pairs after 4300 h. There are no indications of lubricant or system degradation. Two PAO-lubricated bearing pairs are shown in Figure 7 at 4300 h running time. The parts are clearly wet with lubricant, and there are no visual signs of degradation. One pair was reinstalled in the test fixture and has logged more than 21,000 h without degradation in performance.

As a final example, Conley and Bohner observed that Bray 815Z, a linear PFPE oil, was clearly outperformed by a petroleum-based oil (with additives) and by an MoS_2 solid lubricant in testing of an oscillating gimbal assembly.[8] The results are given in Figure 8.

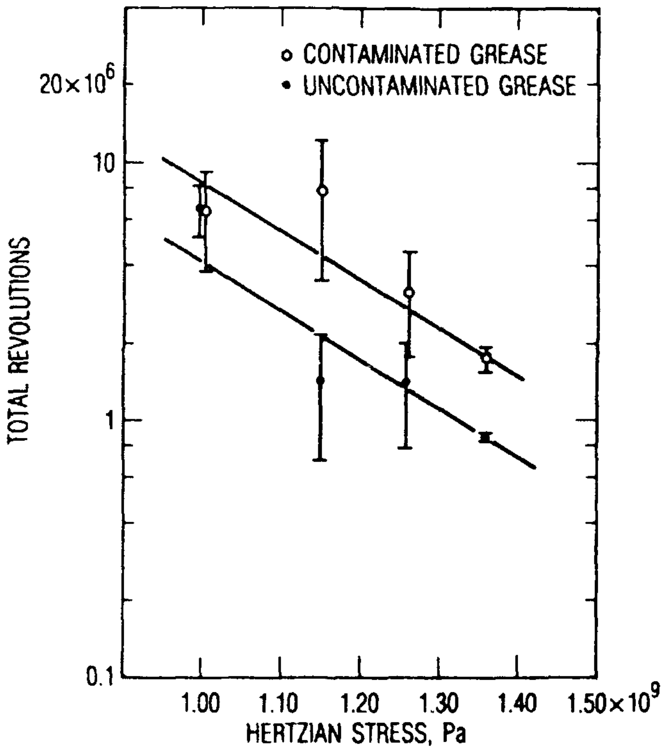


Figure 2. Wear life vs Hertzian stress for contaminated and uncontaminated PFPE grease. (Figure reprinted by permission of the Society of Tribologists and Lubrication Engineers. All rights reserved.)

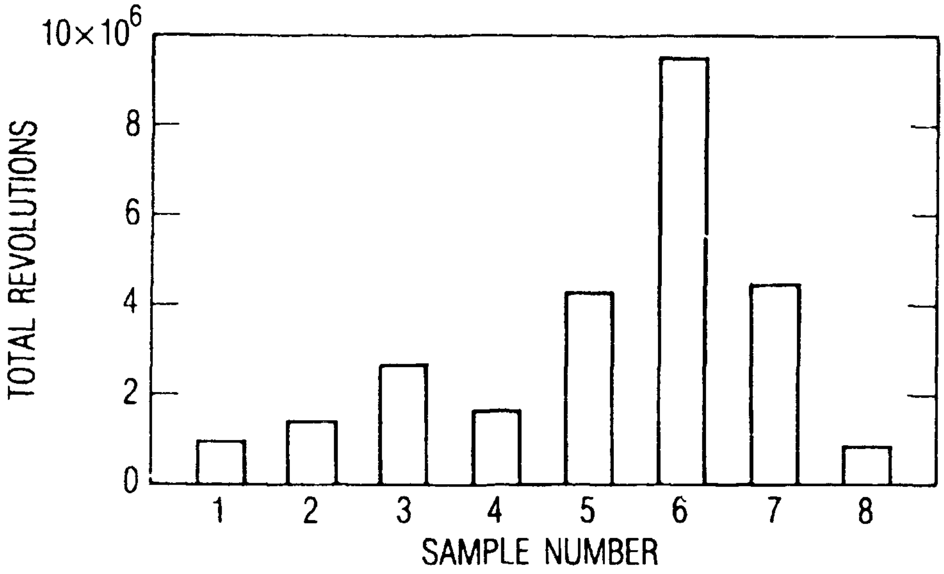


Figure 3. Wear life vs sample number for PFPE and hydrocarbon lubricants. (Figure reprinted by permission of the Society of Tribologists and Lubrication Engineers. All rights reserved.)

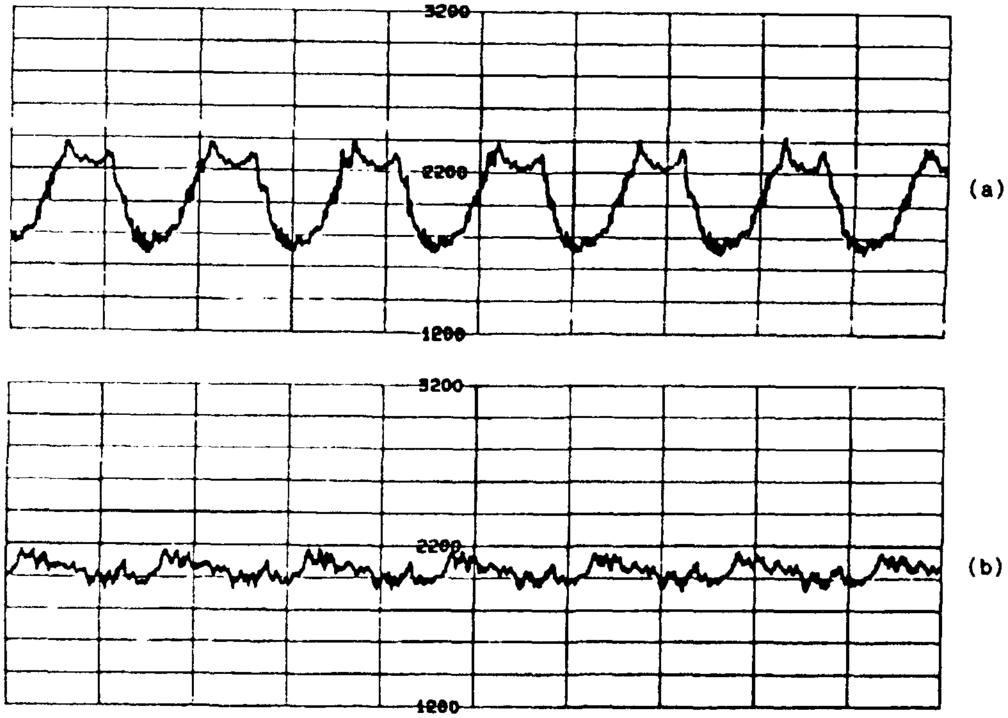


Figure 4. Torque traces for PFPE oil in an oscillatory test. (a) Trace taken at 360 h running time; (b) trace taken at 2350 h. (Figure reprinted by permission of the American Society of Mechanical Engineers. All rights reserved.)

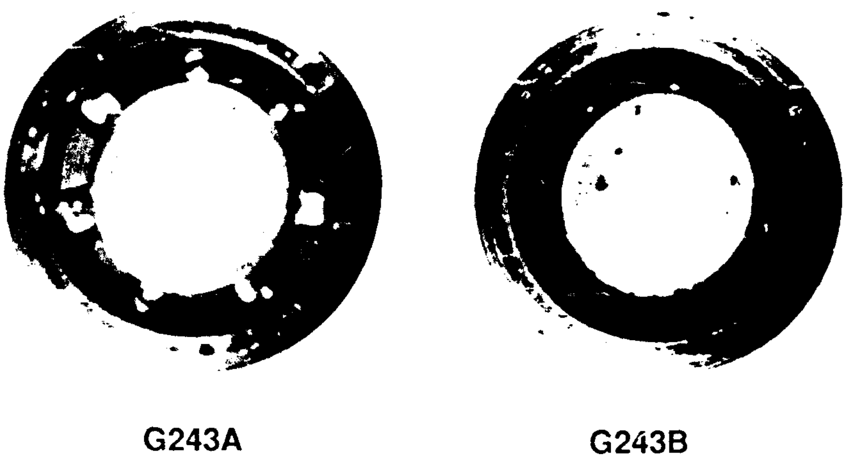


Figure 5. Duplex bearing pair from PFPE oil testing. (Figure reprinted by permission of the American Society of Mechanical Engineers. All rights reserved.)

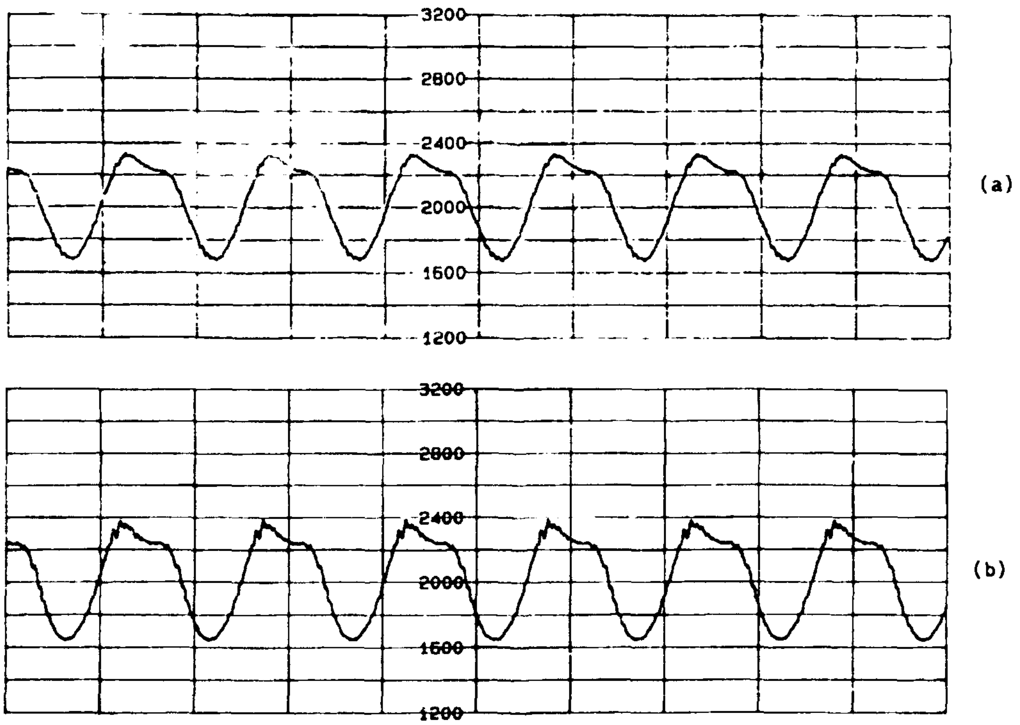


Figure 6. Torque traces for PAO oil in an oscillatory test. (a) Initial trace; (b) trace taken at 4300 h. (Figure reprinted by permission of the American Society of Mechanical Engineers. All rights reserved.)

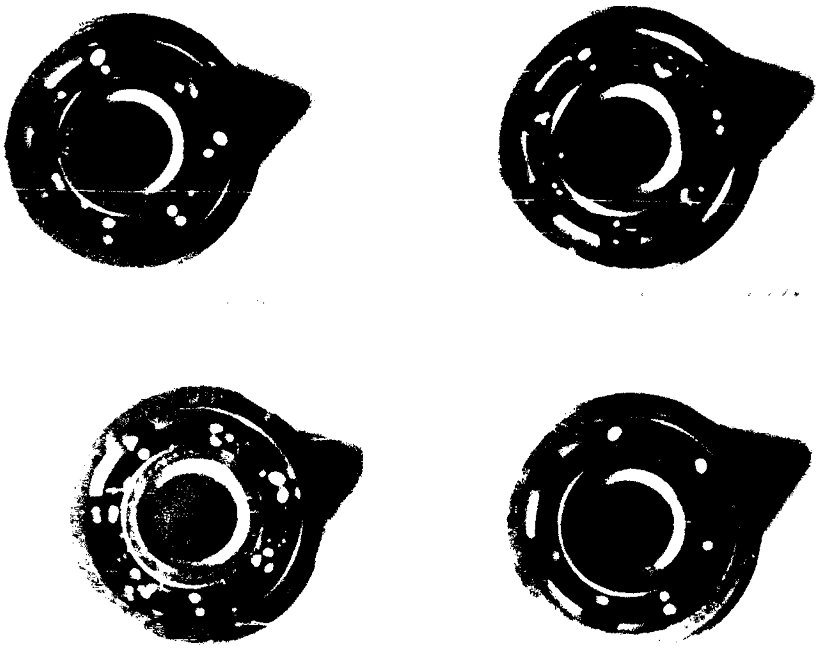


Figure 7. Duplex bearing pairs from PAO oil testing. (Figure reprinted by permission of the American Society of Mechanical Engineers. All rights reserved.)

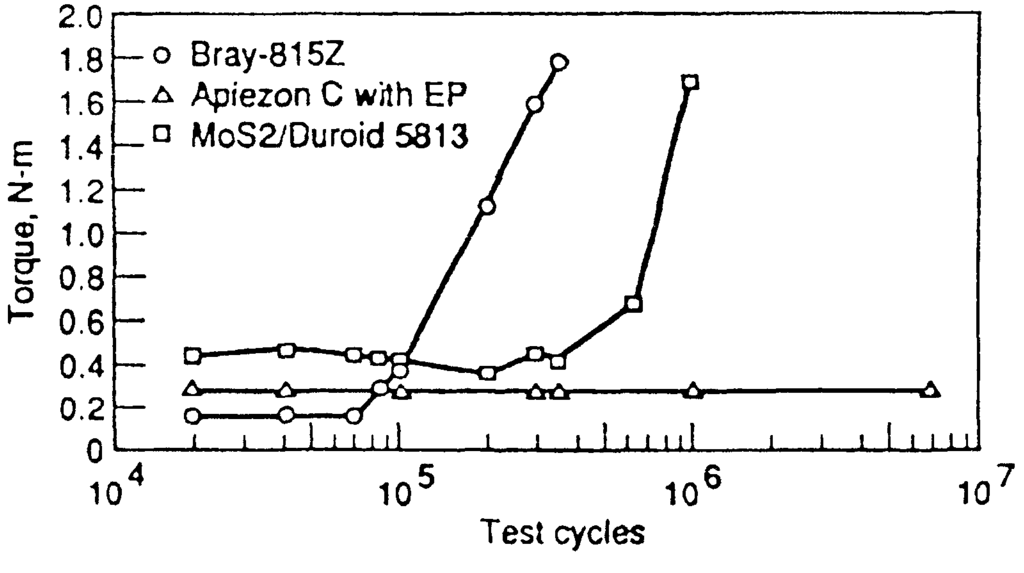
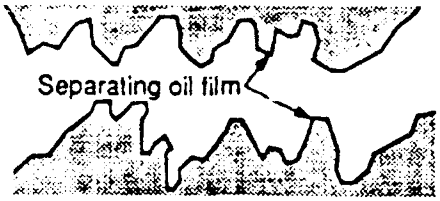


Figure 8. Oscillating gimbal test wear life results. (Figure reprinted by permission of the authors, ref. 8.)

PFPE DEGRADATION HYPOTHESIS AND TESTING

The preceding observations of poor boundary performance led us to propose that the degradation was caused by chemically reactive iron metal generated during the wear process. Figure 9 shows a cartoon of two contacting surfaces under elastohydrodynamic (EHD) and boundary lubrication conditions.[8] In boundary lubrication, the average surface roughness is more than the thickness of the lubricant film, resulting in contact between the asperities on the surfaces. When this occurs, high temperatures are generated in the contact that can "drive" deleterious chemical reactions.

Figure 10 gives our hypothesized scheme for PFPE degradation. Initially, the PFPE oil reacts with iron metal, resulting in the formation of FeF_3. The FeF_3 then catalytically decomposes the PFPE molecules, resulting in polymer formation and loss of volatile degradation products. (In Figure 10, the mechanism is shown stepwise, with ionic intermediates. The mechanism could also be visualized as "concerted," without the need for charged compounds. We have not performed experiments to resolve the fine details of the mechanism.)



a) Elastohydrodynamic Lubrication (EHD)

b) Boundary Lubrication

Figure 9. Lubrication regimes. (Figure reprinted by permission of the authors, ref. 8.)

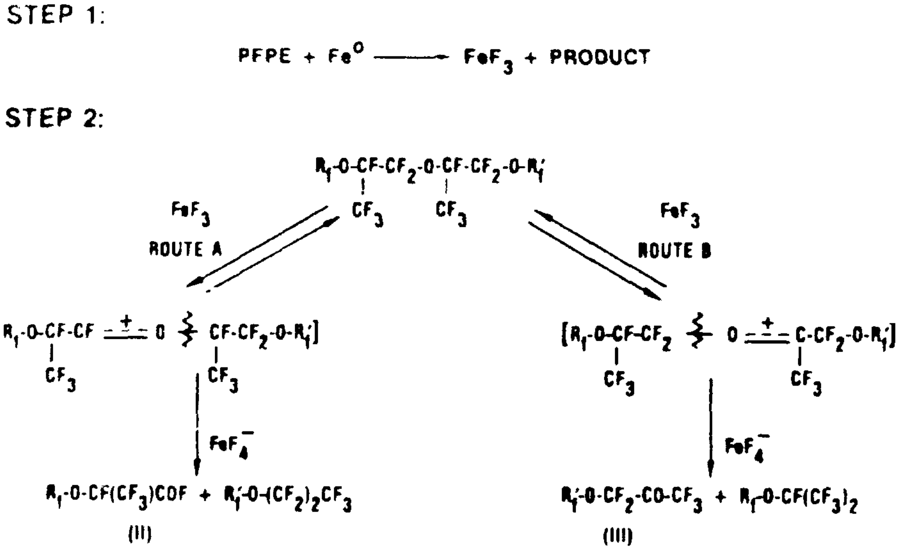


Figure 10. PFPE degradation scheme. (Figure reprinted by permission of the Society of Tribologists and Lubrication Engineers. All rights reserved.)

We attempted to test our hypothesis with a series of experiments. The initial experiments were aimed at demonstrating that FeF_3 does degrade PFPE material.[9] A branched PFPE and FeF_3 were reacted in a nickel-lined autoclave under inert atmosphere at high temperatures. (The inert atmosphere was necesssary to avoid oxidation reactions that have been shown to degrade PFPE.[10]) Figure 11 shows an infrared spectrum of the oil from one of these tests. There are absorption bands that indicate the presence of carbonyl-containing breakdown products, which are predicted by the hypothesized mechanism. Figure 12 shows a high-performance liquid chromatography (HPLC) trace for the same degraded oil. (The HPLC separates the components as a function of molecular weight.) By subtracting the chromatogram of the unused oil from that of the degraded oil, it can be shown that the molecular weight of the oil decreased, consistent with degradation. This subtraction technique is illustrated in the inset in Figure 12. The negative peak at higher molecular weight indicates material that has reacted, and the positive peak at lower molecular weight indicates reaction products.

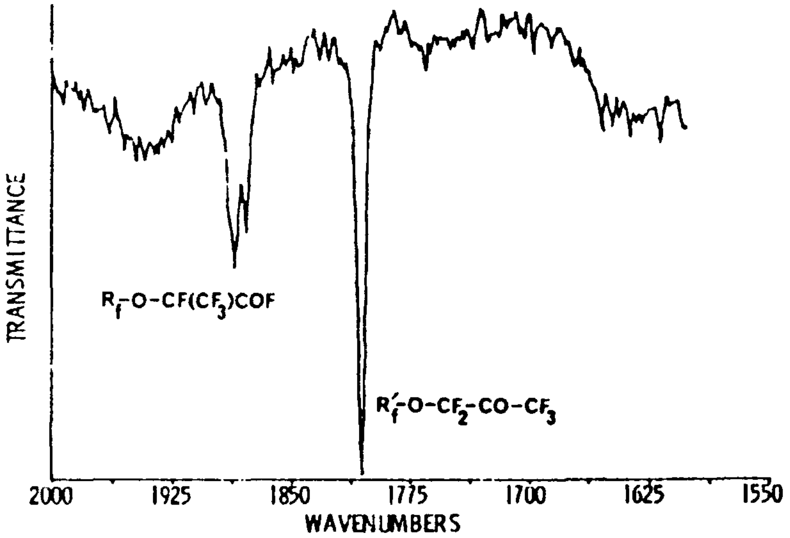


Figure 11. IR spectrum of the products of FeF_3-catalyzed degradation of PFPE oil. (Figure reprinted by permission of Leaf Coppin Publishers. All rights reserved.)

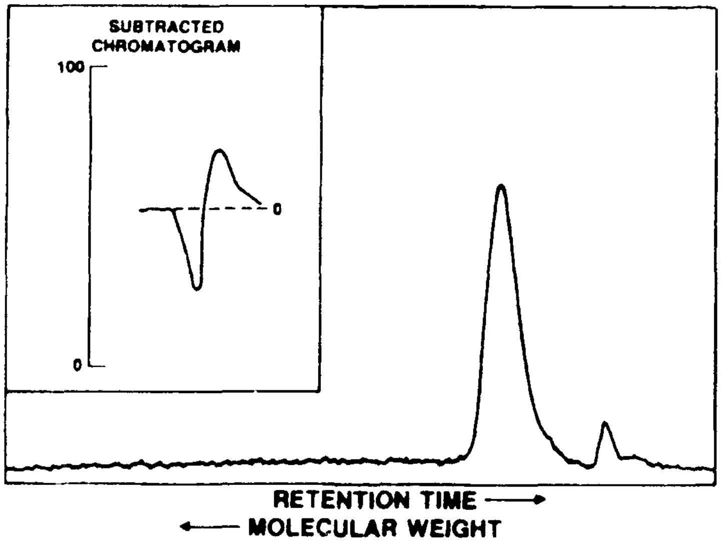


Figure 12. HPLC chromatograms. Typical PFPE oil trace. The inset shows a chromatogram generated by subtracting the trace for the unreacted oil from that of the reacted oil.

These results led us to conclude that the FeF_3-catalyzed degradation occurred at temperatures approximately 30°C below thermal degradation. (The rate vs temperature profiles are shown graphically in Figure 13.) The catalytic reaction corresponds to a high temperature process. However, temperatures are sufficiently high at asperity contacts for the reaction to occur.[11]

Although we had demonstrated that reaction of PFPE fluid with FeF_3 degraded the fluid, we wanted to show that FeF_3 was formed on the contacting surfaces during the wear process. The experimental apparatus is shown in Figure 14.[12] Boundary conditions were achieved through the interaction of a thrust-bearing upper raceway and balls with a flat steel disk. The axes of rotation were not coaxial, resulting in an eccentric motion with a high degree of sliding. This motion produced a wear track ~2 mm wide on the disk (Figure 15) that could be analyzed by various surface spectroscopies. The nominal test conditions were: a speed of 1750 rpm and an axial load of 44.5 N. The temperature was not controlled. X-ray photoelectron spectroscopy (XPS) analysis of the disk wear track indicated that fluoride ion was being generated, as shown in Figure 16. The fluorine peak from the wear track is clearly at lower binding energy than covalent fluorine in PFPE molecules. An Auger electron spectroscopy (AES) fluorine elemental map (Figure 17) showed that the fluorine was limited to the wear track area. Secondary ion mass spectrometry (SIMS) data (Figure 18) showed evidence of FeF^+ ion in the wear track, which indicates the presence of FeF_3 or FeF_2. Thus, iron fluoride compounds were being formed. Unfortunately, we haven't been able to prove definitively that FeF_3 is being formed, although its presence is likely.

There have been other proposed PFPE degradation mechanisms in the literature. Baxter and Hall[13] proposed that interaction of the oxygen in the PFPE molecules with exposed iron resulted in cleavage of the molecules and subsequent polymerization.

Zehe and Faut[14] have proposed that initial reaction of the PFPE with iron oxides on the metal surfaces results in PFPE degradation and concomitant formation of FeF_3, which further catalyzes the degradation. They also concluded that the linear PFPE molecules are more reactive because they contain chemically labile -O-CH_2-O- acetal units.

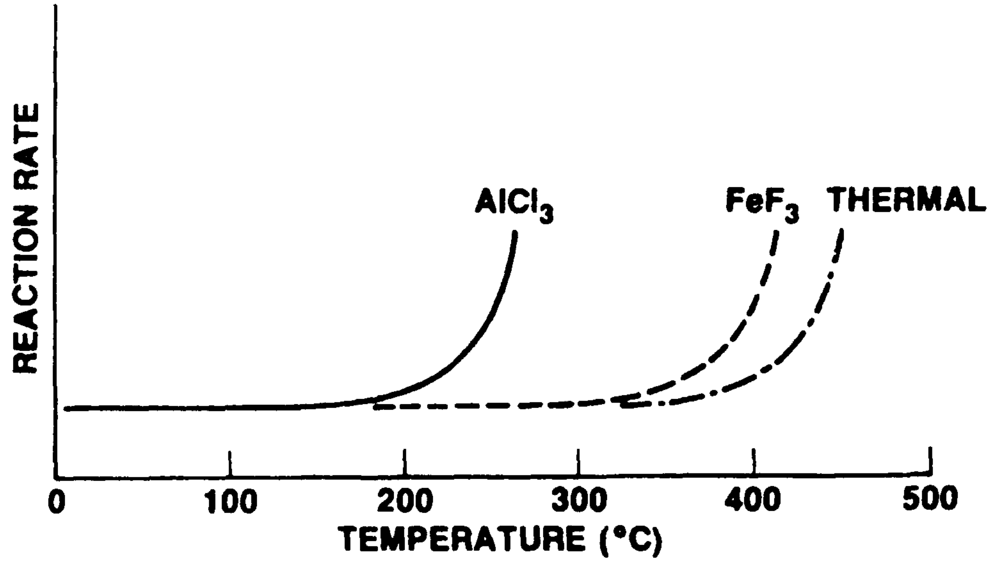


Figure 13. PFPE degradation rate vs temperature.

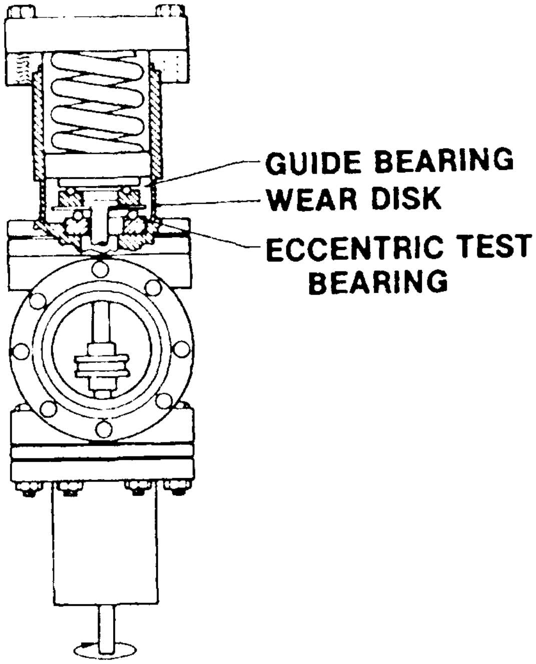


Figure 14. Eccentric thrust bearing-on-disk wear test apparatus. (Figure reprinted by permission of the Society of Tribologists and Lubrication Engineers. All rights reserved.)

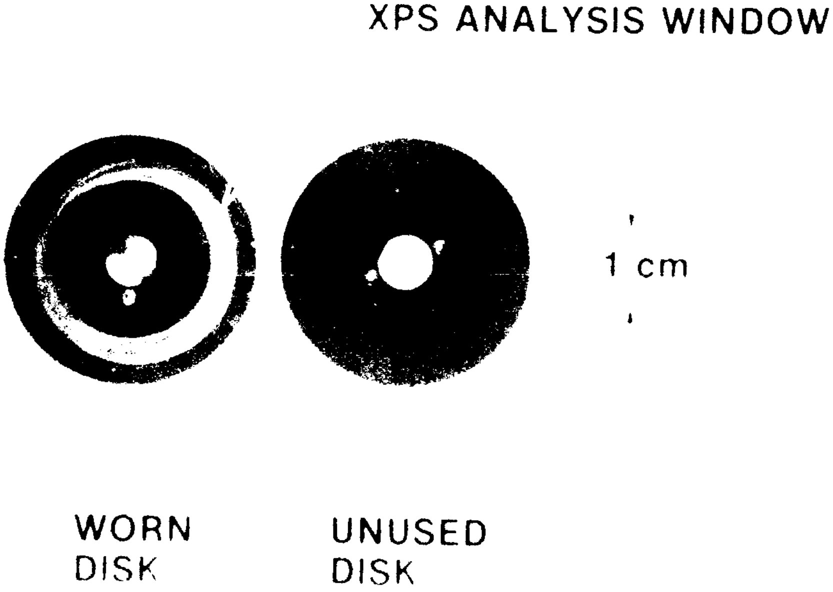


Figure 15. Typical wear track generated on wear disk.

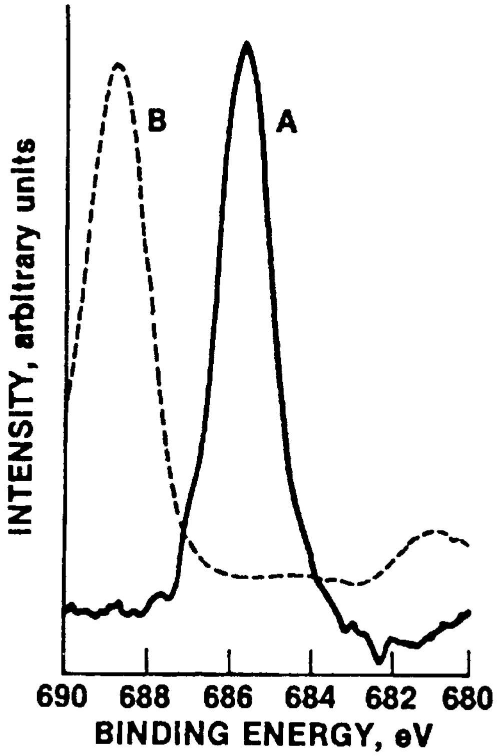


Figure 16. Fluorine XPS spectra: A - Wear disk fluoride ion spectrum; B - PFPE covalent fluorine spectrum. (Figure reprinted by permission of the Society of Tribologists and Lubrication Engineers. All rights reserved.)

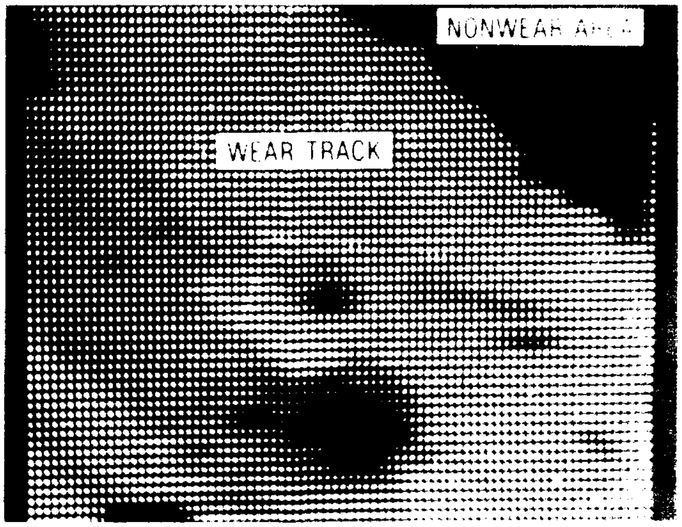
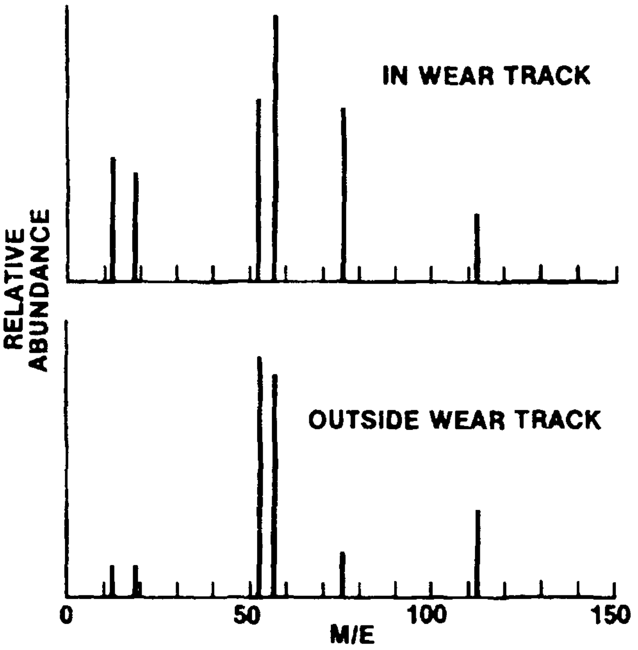


Figure 17. AES fluorine elemental map at edge of wear track. (Figure reprinted by permission of the Society of Tribologists and Lubrication Engineers. All rights reserved.)

Mori and Morales,[15] in a clever experiment, used a mass spectrometer to monitor PFPE degradation products resulting from a sliding-type interaction. They determined that, when sliding stopped, the concentrations of specific degradation products (e.g., CF_2O) decayed slowly, whereas the concentrations of other products (CF_3) decayed quickly. The results are shown in Figure 19.



M/E	ION	RELATIVE ABUNDANCE
12	C^+	47
19	F^+	40
52	Cr^+	68
56	Fe^+	100
75	FeF^+	65
112	Fe_2^+	25

M/E	ION	RELATIVE ABUNDANCE
12	C^+	13
19	F^+	13
52	Cr^+	100
56	Fe^+	93
75	FeF^+	18
112	Fe_2^+	37

Figure 18. SIMS spectra of wear disk.

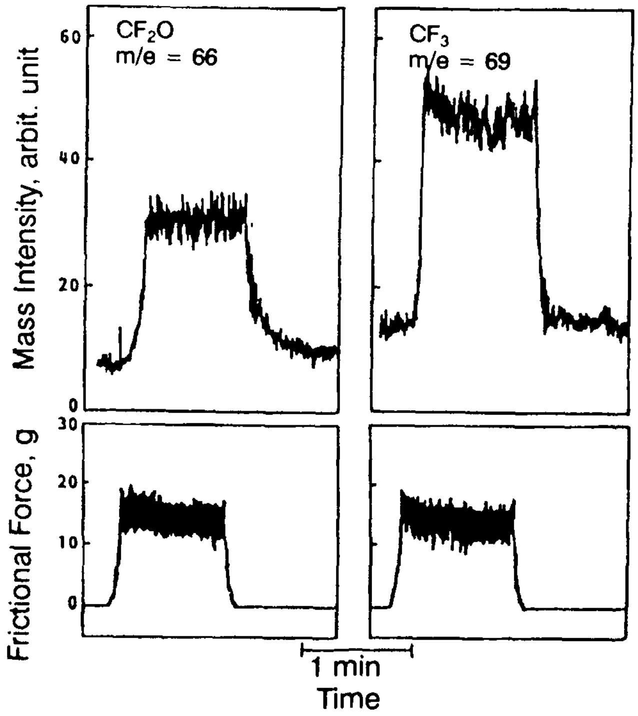


Figure 19. Tailing behavior of COF_2 evolution due to catalytic reactions. (Figure reprinted by permission of Elsevier Sequoia Publishers. All rights reserved.)

According to Mori and Morales, these results indicate that interaction of PFPE with exposed metal initiates degradation and that catalytic degradation, due to FeF_3, continues after sliding has ceased. Their results also support the conclusion that the acetal linkages in the linear PFPE structure [structure (I)] are more reactive than other functional groups in the PFPE molecules, leading to more facile degradation of the linear PFPEs.

All the proposed degradation mechanisms have a common thread: interaction of PFPE with exposed iron metal results in the degradation of the PFPE material. The question to ask is: Can something be done to reduce or eliminate this interaction and prolong the lifetime of PFPE lubricated systems when boundary conditions are present?

PFPE SYSTEM IMPROVEMENT POSSIBILITIES

There are two reasonable approaches to improving the performance of the PFPE materials: the use of antiwear additives and the elimination of interactions that expose iron metal on the surfaces.

Hydrocarbon lubricants use antiwear additives to prolong their performances under boundary conditions. Without these additives, their performances would be considerably poorer. With the PFPE materials, additive solubility is an important consideration. An unfortunate characteristic of PFPE oils is that they are very poor solvents; i.e., very few types of compounds will dissolve or become miscible. For example, the only solvent that is recommended for PFPEs is 1,1,2-trichloro-1,2,2-trifluoroethane (Freon TF). The poor solvent characteristics are surely related to the lack of chemical reactivity that these materials exhibit.

There are efforts under way to synthesize additives for the PFPE fluids. One report in the literature has indicated that improved four-ball wear scar and traction test performance was achieved through the use of a recently synthesized antiwear additive.[16] However, to date, known successes have been limited.

Limiting or eliminating the exposure of iron metal during wear can be achieved through the use of ceramic hard-coated or solid ceramic ball-bearing components. For example, significant improvement in the wear characteristics of instrument bearings under EHD conditions have been achieved through the use of TiC hard-coated and Si_3N_4 solid components with petroleum-based or synthetic hydrocarbon lubricants.[17-20]

We performed a series of experiments to test the effect of ceramic surfaces on wear life.[21] The apparatus shown in Figure 14 was used. The tests were conducted with the materials combinations given in Table 1. The conditions were as follows: speed, 1750 rpm; axial load, 89-200 N; ambient pressure, $< 1.3 \times 10^{-3}$ Pa; and lubricant quantity, 10 µL. The temperature was not controlled in the testing.

Table 1. Wear Test Materials Combinations

Disks	Balls	Raceways
440C	440C	52-100
440C	Si_3N_4	52-100
TiN	TiN	TiN

The results of the experiments are shown in Figures 20 and 21. In Figure 20, the wear life (total revolutions to failure) for the various materials combinations is shown plotted vs axial load. The data for 440C balls and disk lie on essentially the same line as those for the Si_3N_4 balls and 440C disk, whereas the data for the TiN hard-coated components lie higher on the graph. The apparent lack of wear enhancement in the hybrid system is the result of the higher modulus of Si_3N_4, which results in higher Hertzian stress than for the 440C system at the same applied load. We feel that plotting the data vs applied load biases the results because it is the Hertzian stress that influences the chemistry at the contact. However, the graph in Figure 20 has significance in an engineering sense; i.e., if the applied load is fixed, the TiN hard-coated components are clearly the appropriate choice for lifetime enhancement.

Figure 21 shows a plot of wear life vs Hertzian stress. The data for the Si_3N_4/440C hybrid system are now grouped with the TiN data. Thus, these data indicate that enhancements in system lifetime on the order of 5 to 10 are attainable.

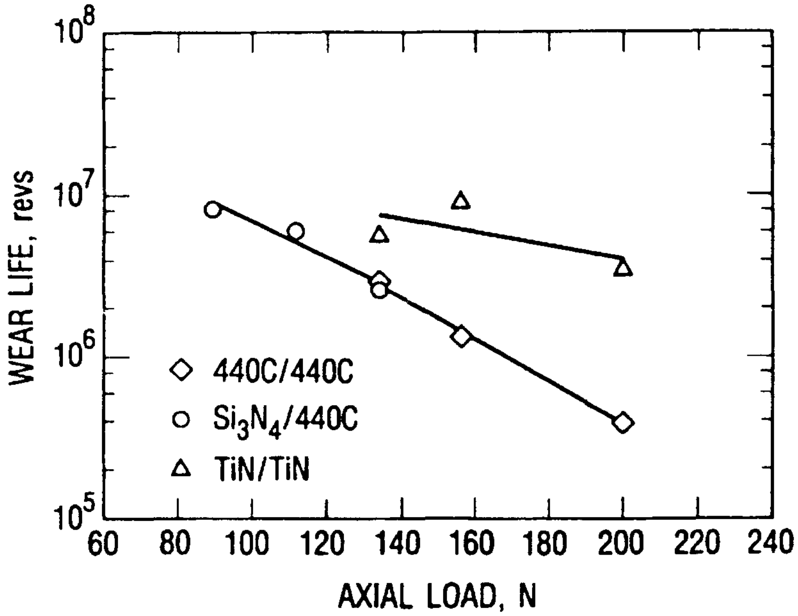


Figure 20. Wear Life as a function of axial load.

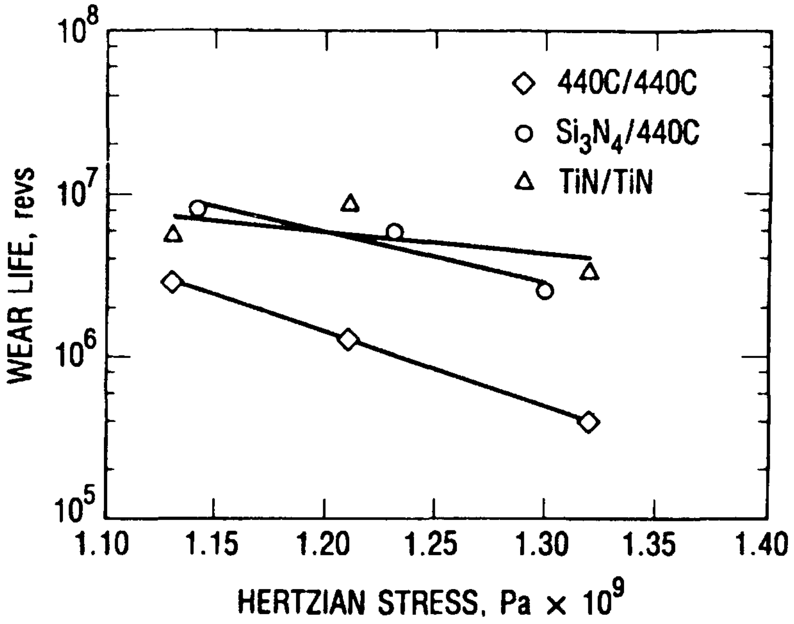


Figure 21. Wear life as a function of Hertzian stress.

When the experiment was conceived, our initial expectation was that the average wear life values for the TiN-coated system would be significantly larger than for the Si_3N_4/440C system. In the latter system, some metallic iron would be expected to be exposed because one surface is steel, resulting in some catalysis, whereas for the TiN system, no catalysis would be expected. We believe that the lack of differentiation in the two systems is the result of a kinetic effect.

We have previously demonstrated that the FeF_3-catalyzed degradation of the PFPE fluid became kinetically significant at 350°C and that the rate of thermal degradation was not comparable until 380°C.[9] If we use the back-of-the-envelope approximation that the degradation rate doubles for each 10°C increase in temperature, we arrive at the estimate that thermal degradation would be slower than catalyzed degradation by a factor of 8 at 350°C. We conclude that, by reducing or eliminating the catalyzed degradation pathway, the limiting reaction becomes thermal degradation. To the first order, thermal degradation is not influenced by choice of materials. This conclusion implies that the maximum improvement that could be achieved under the test conditions is approximately a factor of 8, which is consistent with the lifetime increases measured in our testing. This point is illustrated graphically in Figure 22. At low iron concentrations at the surface, the reaction rate is controlled by the rate of thermal degradation. As the concentration of iron increases, the catalytic and total degradation rate also increase and catalysis becomes dominant.

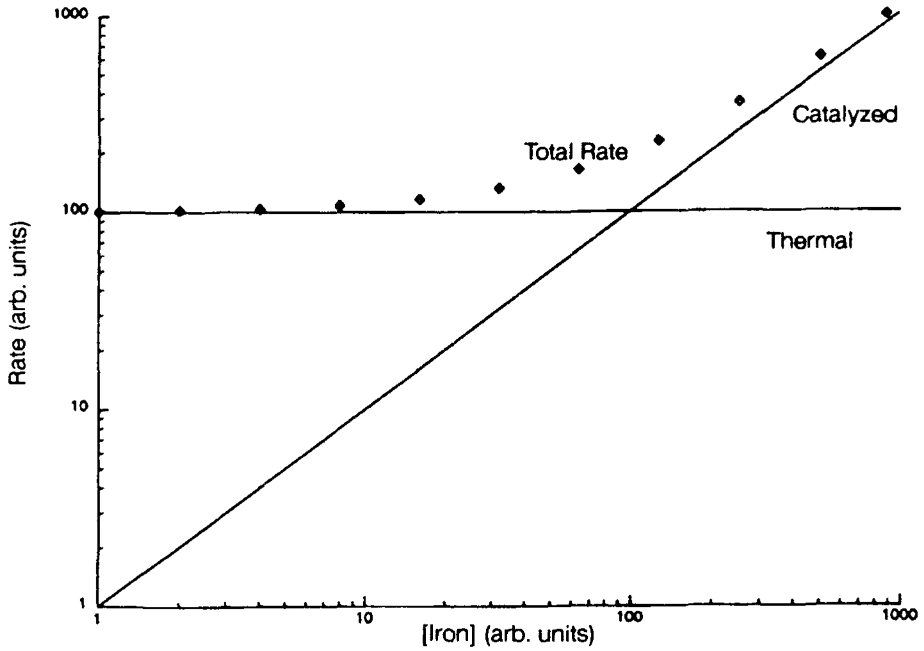


Figure 22. PFPE degradation rate.

CONCLUSIONS

The PFPE family of lubricants offers outstanding properties that make them very attractive for spacecraft applications. Their poor boundary-conditions performance can be significantly improved through the use of antiwear additives, once they are proven, and through the use of ceramic hard-coated or solid ball-bearing components. In our studies with ceramic surfaces, a factor of 10 improvement in wear life has been observed. This could mean an extension of wear life from 1 to 10 years in an actual application, which is a dramatic improvement.

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